

TRANSMITTAL LETTER TO THE UNITED STATES  
DESIGNATED/ELECTED OFFICE (DO/EO/US)  
CONCERNING A FILING UNDER 35 U.S.C. 371

U.S. APPLICATION NO. (If known, see 37 CFR 1.5)

INTERNATIONAL APPLICATION NO.

INTERNATIONAL FILING DATE

PRIORITY DATE CLAIMED

PCT/EP00/00909

February 4, 2000

February 4, 1999

TITLE OF INVENTION

METHOD FOR dimensionally sintering ceramics

APPLICANT(S) FOR DO/EO/US

HAUPTMANN, Holger; BURGER, Bernd; SCHNAGL, Robert; WAGNER, Ingo

Applicant herewith submits to the United States Designated/Elected Office (DO/EO/US) the following items and other information:

1. ☒ This is a **FIRST** submission of items concerning a filing under 35 U.S.C. 371.
2. ☐ This is a **SECOND** or **SUBSEQUENT** submission of items concerning a filing under 35 U.S.C. 371.
3. ☒ This express request to begin national examination procedures (35 U.S.C. 371(f)) at any time rather than delay examination until the expiration of the applicable time limit set in 35 U.S.C. 371(b) and PCT Articles 22 and 39 (1).
4. ☐ The US has been elected by the expiration of 19 months from the priority date (Article 31).
5. ☒ A copy of the International Application as filed (35 U.S.C. 371(c)(2))
- a. ☒ is transmitted herewith (required only if not transmitted by the International Bureau). WO 00/46166
- b. ☒ has been transmitted by the International Bureau.
- c. ☐ is not required, as the application was filed in the United States Receiving Office (RO/US).
6. ☒ An English language translation of the International Application as filed (35 U.S.C. 371(c)(2)).
- a. ☒ is transmitted herewith.
- b. ☐ has been previously submitted under 35 U.S.C. 154(d)(4)
7. ☒ Amendments to the claims of the International Application under PCT Article 19 (35 U.S.C. 371(c)(3)).
- a. ☒ are transmitted herewith (required only if not transmitted by the International Bureau).
- b. ☐ have been transmitted by the International Bureau.
- c. ☐ have not been made; however, the time limit for making such amendments has NOT expired.
- d. ☐ have not been made and will not be made.
8. ☒ An English language translation of the amendments to the claims under PCT Article 19 (35 U.S.C. 371(c)(3)).
9. ☐ An oath or declaration of the inventor(s) (35 U.S.C. 371(c)(4)).
10. ☐ An English language translation of the annexes of the International Preliminary Examination Report under PCT Article 36 (35 U.S.C. 371(c)(5)).

Items 11. to 20. below concern document(s) or information included:

11. ☒ An Information Disclosure Statement under 37 CFR 1.97 and 1.98-International Search Report (PCT/ISA/210) w/ 6 documents
12. ☐ An assignment document for recording. A separate cover sheet in compliance with 37 CFR 3.28 and 3.31 is included.
13. ☒ A **FIRST** preliminary amendment.
14. ☐ A **SECOND** or **SUBSEQUENT** preliminary amendment.
15. ☐ A substitute specification.
16. ☐ A change of power of attorney and/or address letter.
17. ☐ A computer-readable form of the sequence listing in accordance with PCT Rule 13ter.2 and 35 U.S.C. 1.821-1.825.
18. ☐ A second copy of the published international application under 35 U.S.C. 154(d)(4).
19. ☐ A second copy of the English language translation of the international application under 35 U.S.C. 154(d)(4).
20. ☒ Other items or information:
- 1.) Certified Copy of the translation of the International Application
- 2.) Six (6) sheets of Formal Drawings

/cqc

PATENT  
0475-0193P

## IN THE U.S. PATENT AND TRADEMARK OFFICE

Applicant: HAUPTMANN, Holger et al. Conf.:  
Int'l. Appl. No.: PCT/EP00/00909  
Appl. No.: New Group:  
Filed: August 3, 2001 Examiner:  
For: METHOD FOR DIMENSIONALLY SINTERING CERAMICS

PRELIMINARY AMENDMENT**BOX PATENT APPLICATION**

Assistant Commissioner for Patents  
Washington, DC 20231

August 3, 2001

Sir:

The following Preliminary Amendments and Remarks are respectfully submitted in connection with the above-identified application.

**AMENDMENTS****IN THE SPECIFICATION:**

Please amend the specification as follows:

Before line 1, insert --This application is the national phase under 35 U.S.C. § 371 of PCT International Application No. PCT/EP99/00909 which has an International filing date of February 4, 2000, which designated the United States of America and was not published in English.--

**IN THE CLAIMS:**

Please amend the claims as follows:

13. (Amended) Process according to claim 1 or 2, the preform containing aluminum oxide, zirconium oxide or mixed oxides of both.

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**REMARKS**

The specification has been amended to provide a cross-reference to the previously filed International Application. The claims have also been amended to delete the improper multiple dependency of claim 13 and to place the application into better form for examination. Entry of the present amendment and favorable action on the above-identified application are earnestly solicited.

Attached hereto is a marked-up copy of the changes made to the application by this Amendment.

If necessary, the Commissioner is hereby authorized in this, concurrent, and future replies, to charge payment or credit any overpayment to Deposit Account No. 02-2448 for any additional fees required under 37 C.F.R. § 1.16 or under 37 C.F.R. § 1.17; particularly, extension of time fees.

Respectfully submitted,

BIRCH, STEWART, KOLASCH & BIRCH, LLP

By 

Andrew D. Meikle, #32,868

ADM/cqc  
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Attachment: Version With Markings Showing Changes Made

(Rev. 01/22/01)

**VERSION WITH MARKINGS SHOWING CHANGES MADE**

The specification has been amended to provide cross-referencing to the International Application.

The claims have been amended as follows:

13. (Amended) Process according to [one of claims 1 to 12]claim 1 or 2, the preform containing aluminum oxide, zirconium oxide or mixed oxides of both.

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Process for the dimensionally-true sintering of ceramics

The invention relates to a process for the dimensionally-true sintering of free-form flat ceramics. In particular, the invention relates to a process for dimensionally-true sintering of dental prostheses prepared from dental ceramics.

Because of their physical properties, ceramics are much valued in the construction of high-quality pre-shaped parts, for example dentures and are therefore ever more widely used. Upon sintering of ceramic materials, a volume reduction (shrinkage) always takes place. During the firing process parts of the object to be sintered perform a movement relative to a rigid, non-movable firing base. With filigree works which are used in particular in the field of dentures, the free movability is hampered by minor hooking effects on the firing base, a considerable deformation of the object thereby occurring. This state of affairs is particularly critical with bridges which are composed for example of two caps and a crosspiece connecting them: a deformation of the original geometry of the bridge occurs which has a very adverse effect on the accuracy of fit of the prosthetic work.

Usually, powders are used to reduce the friction between firing material and firing base. At higher sinter temperatures, however, either reactions between powder and firing material, or a caking of the powder fill caused by the development of sinter necks, occurs. In both cases, this can lead to the effect described above and thus to the unusability of the firing material. Because of the preform's own weight, deformation of the

preform structures can also occur in systems which display super-elasticity. This effect occurs with bridges in particular.

5 It is known from DD-121 025 to fire mouldings formed bodies on firing bases which are coated with molybdenum. Such processes are in principle unsuitable for high-quality ceramic workpieces, as a contamination of the ceramic by metal parts occurs because of diffusion  
10 processes.

The object of this invention is to provide a process which allows a dimensionally-true sintering of ceramic pre-shaped items.

15 This object is achieved according to the invention by resting the firing material during the sintering on supporting materials, not coated with metal, which adapt independently to the shrinkage dimensions which occur  
20 during the firing process or allow a contact-free support of the pre-shaped items.

The supporting materials according to the invention can be designed in completely differently ways. The design  
25 shapes can in principle be divided into the following groups:

I. Resting of the firing material on movable supporting materials which can be composed of any material, for  
30 example based on sintered aluminium oxide, which is inert vis-à-vis the firing process and does not result in adhesion to the firing material and does not contaminate the latter.



II. Resting of the firing material on supporting materials which have the same physical properties as the firing material itself. Preferably, the support is composed of the same material as the firing material, for example based on zirconium oxide or aluminium oxide.

III. Resting of the firing material on supporting materials which have very different physical properties to the firing material itself, in which case a contamination or bonding of the firing material with the supporting material must not be possible.

IV. Resting of the firing material on supporting materials which allow a contact-free support.

Possible versions of group I of the processes according to the invention are reproduced in the following.

In principle, with this process variant, the firing material rests on a movable support. These supports are to be housed in a base, attached via a suspension means or designed so that they require no attachment.

In particular, the following versions are suitable as base:

- Fire-proof firing wadding, for example a fleece made of aluminium oxide, containing  $\text{SiO}_2$ .
- Fire-proof firing sand, for example corundum.
- Divided structures, open to the top, for example honeycombed structures, in which a tipping of the movable support within the framework of the firing

process is possible in simple manner, for example those made of mullite.

- Fire-proof packing materials which have sufficient flexibility to yield to the forces which occur during the firing process, for example those made of aluminium oxide.
- Fire-proof base plates which have the same shrinkage as the firing material, for example those made of aluminium oxide.

The following versions in particular are suitable as suspension means:

- Suspension via fixed-mounted hooks, the firing material being fitted at a suitable position onto at least two hooks made of fire-proof material, for example aluminium oxide, and the hooks approaching each other through the forces occurring during the firing process.

Figure 1 shows by way of example the attachment of two S-shaped hooks (X) at a fixed position (Y) within a firing chamber (Z), the firing material (A) already being fitted onto the hooks. The design of the firing material is only represented schematically here and at all other points and is not in any way to be understood as limitative.

- Suspension via movably applied hooks, the firing material being fitted at a suitable position onto at least two hooks made of fire-proof material, for example aluminium oxide, and the hooks being attached movable inside or outside the firing chamber.

Figure 2 shows by way of example the attachment of two S-shaped hooks (X) inside the firing chamber (Z), each of the hooks being freely movable on a track (S), for example over rollers, and thus being able to yield to the forces which occur during the firing process and the firing material (A) already being fitted onto the hooks.

The hooks can also be suspended in a bar-shaped track structure (B) as shown in Figure 3. The structure consists of vertical elements of (B) and horizontal elements of (B) which permit a suspension of the hooks (X) which support the firing material (A).

In principle, each method of attaching two hooks flexibly at a suitable height can be used.

Figure 4 shows by way of example the attachment of two hooks (X) outside the firing chamber (Z), each of the hooks being freely movable on a sliding bearing (G) and thus being able to yield to the forces which occur during the firing process. As the movable supports are located outside the firing chamber, the process is preferably applied such that the firing chamber is screened from the supports via a suitable heat insulator (W). This variant of the process according to the invention can also be improved in that the movement of the hooks in the sliding bearings does not take place exclusively through the forces occurring during the firing process, but in that the change of position of the hooks

in the sliding bearings that is necessary for a force equalization is established by a mechanical, electronic and/or optical scanning device (V), and carried out mechanically for example (principle of the tangential record-player).

- Within the meaning of this invention, the term suspension is also taken to mean devices which use the same principle as described previously, except that the sliding bearings are attached below the firing material, these being able to be located inside or outside the firing chamber.

Figure 5 shows by way of example the attachment of two props (T) for the firing material, the props being freely movable on sliding bearings (G) outside the firing chamber (Z) and thus being able to yield to the forces which occur during the firing process. A heat insulator (W) can be advantageous here just as a mechanical, electronic and/or optical scanning device (V) which establishes and carries out, for example mechanically, the change in position of the hooks in the sliding bearing necessary for a force equalization.

As supports or props, the following versions in particular are suitable:

- Rods which have a cross-section which allows a minimal contact surface with the firing material, for example circular, elliptical, rectangular, in particular square and rhomboid, convex, concave,

triangular, U-shaped cross-sections, the rods being able to be hollow or solid; the rods can be arranged to stand vertically or lie horizontally.

- 5 • Supporting materials which have a tip which allows a minimal contact surface with the firing material, for example arrow-shaped, pyramid-shaped, conical supports which can be hollow or solid.

10

The following versions in particular are suitable as supporting materials which require no suspension and no attachment:

- 15 • Drop-shaped bodies (tumblers) which, because of their weight distribution, come to rest in such a way that the tip of the body is perpendicular to the bearing surface at the beginning of the firing process. During the firing process, the tips of  
20 the bodies move towards each other because of the shrinkage forces which occur.

The named supports, rollers, suspensions or props can be composed of all refractable metals, metal oxides, metal  
25 carbides and their mixtures, in particular of  $\text{Al}_2\text{O}_3$ ,  $\text{MgO}$ ,  $\text{ZrO}_2$ ,  $\text{SiO}_2$ , cordierite,  $\text{SiC}$ ,  $\text{WC}$ ,  $\text{B}_4\text{C}$ ,  $\text{W}$ ,  $\text{Au}$ ,  $\text{Pt}$ .

Figures 6 and 7 show further embodiments for group I.

- 30 Figure 6 shows the placing of a bridge (1) on rods (2) which are housed flexibly inside so-called firing wadding (3). During the sintering process, the rods (2) can move independently in the direction of the shrinkage without tipping or deforming the bridge (1).

Figure 7 shows another version. The prosthetic work (1) is laid on a roller-shaped structure (2), the distances between the rollers adjusting independently during the firing process. The rollers are housed on suitable suspensions or props, for example in T- or U-shape.

With small ceramic pre-shaped items, individual or some few supports and/or props are sufficient. With large pre-shaped items, several to very many supports and/or props are required which are optionally housed such that their bearing points can adapt to the shape of the pre-shaped item to be sintered.

Possible versions for group II of the processes according to the invention are reproduced in the following.

- The supporting pins (3) required during the milling of the work piece (1) are left in place after the milling process so that they serve as a stable multipoint support on a level firing base with the same shrinkage behaviour. The supporting device according to the invention consists in this case of the supporting pins (3) and a plane firing base made of material with the same shrinkage behaviour as the prosthetic work, preferably of the same material as the prosthetic work. Particularly preferably, a plane surface (5) is simultaneously left on the pre-shaped body during the milling process in addition to the holding pins (3), the preform (2) having to be correspondingly large in size. The supporting pins (3) are separated after the sintering in order to obtain the desired pre-shaped body. The device for

the process according to the invention is placed on a fire-proof firing base (6) for example using a pourable fill material (4) or suitable support and/or props. Figure 8 is intended to explain this version in more detail.

• Cutting through supporting pins even before the sintering, fitting the remainder of the original preform (2), which after milling corresponds to a negative mould (3) of the prosthetic work, onto a plane firing base (5) using separating powder (4). Coating of the inside of the negative mould (3) likewise with separating powder (4) and laying-up of the prosthetic work (1) to be fired. The preform remainder (3) serves together with the separating powder (4) as a supporting device according to the invention (Figure 9). The device for the process according to the invention is placed on a fire-proof firing base (6), for example using a pourable fill material (4) or suitable supports and/or props. Surprisingly, the development of sinter necks within the fill, comprising separating powder, does not take place.

All refractable metals, metal oxides, metal carbides and their mixtures, in particular  $\text{Al}_2\text{O}_3$ ,  $\text{MgO}$ ,  $\text{ZrO}_2$ ,  $\text{SiO}_2$ , cordierite,  $\text{SiC}$ ,  $\text{WC}$ ,  $\text{B}_4\text{C}$ , can be used as separating powders.

Figure 10 shows the firing material (A) resting on two Y-shaped supports (B). Two holding pins (H) are attached to the firing material (A) which are either produced during the shaping process or attached to the firing material after the shaping process. The supporting pins preferably

consist of the same material as the firing material, particularly preferably they are made from the same preform. Depending on the version (different or same material), this type of placement is to be allocated to group I or II. In principle, mixed versions can also be considered which are to be allocated simultaneously to the different groups.

Possible versions for group II of the processes according to the invention are reproduced in the following.

- In principle, all supporting materials are suitable which have very different physical properties to the firing material itself. A contamination or bonding of the firing material with the supporting material must be excluded. The melting point of such materials preferably lies below 1450°C, particularly preferably below 1400°C. The density preferably lies somewhat above that of the firing material so that the latter can float on the supporting material. Metals or metal alloys, for example gold, can also be suitable.

Possible versions for group IV of the processes according to the invention are reproduced in the following.

- The firing material rests on a gas jet, the firing material floating contact-free above the floor of the firing chamber. Control apparatuses which direct the gas jet so that the firing material can float in stable manner are also advisable. Preferably, the gases used are non-reactive gases, for example inert gases. To optimize the gas streams, control systems of all types can be used.



• The firing material rests on magnetic fields, at least one magnetic substance being attached at a suitable point in the firing material, the firing base itself or a corresponding bearing surface also being magnetic and the polarity of the two magnetic fields being identical. A magnetic design of parts of the firing material itself is also possible.

Figure 11 shows the firing material (A) resting on a magnetic field which is generated by the magnetic bases or pre-shaped parts (M), the polarity of the magnets having to be such that the firing material floats away from the base. The whole device is located in the firing chamber (Z). Preferably, permanent magnets are used as magnets (M). The use of electromagnets or a mixed use of the magnet types which can be considered is also possible.

Figure 12 shows the firing material (A) resting on gas streams (L), the latter exiting through a base plate provided with throughflow openings. The devices are located inside the firing chamber (Z), it being also advantageous if the floor of the firing chamber is already provided with the throughflow openings and the control and generation of the gas streams takes place outside the firing chamber.

ART 34 AMDT

New Patent Claims

1. Process for the dimensionally-true sintering of ceramic pre-shaped items, the firing material resting during the sintering on supporting materials, not coated with metal, or consisting of metal molten at the sinter temperature, which adapt independently to the shrinkage dimensions which occur during the firing process or allow a contact-free support of the pre-shaped items.
2. Process according to claim 1, the pre-shaped items being ceramic dental prostheses.
3. Process according to one of claims 1 or 2, the firing material resting on movable supporting materials which can be composed of any material which is inert vis-à-vis the firing process and does not result in adhesion to the firing material and does not contaminate the latter.
4. Process according to claim 3, the supporting materials being developed as vertically standing or horizontally lying hollow or solid rods and having a cross-section which allows a minimal contact surface with the firing material.
5. Process according to claim 3, the supporting materials having a tip which allows a minimal contact surface with the firing material, and being hollow or solid.
6. Process according to one of claims 1 or 2, the firing material resting on supporting material which

has the same physical properties as the firing material itself.

7. Process according to claim 6, supporting material and firing material being prepared from the same preform.
8. Process according to claim 7, the firing material being connected to a plane surface via supporting pins which are cut through after sintering.
9. Process according to claim 7, the firing material resting in the negative mould obtained from the preform through the milling process on a pourable fill material or on suitable supports and/or props.
10. Process according to one of claims 1 or 2, the firing material resting on supporting material which has very different physical properties to the firing material itself, where a contamination or bonding of the firing material with the supporting material must not be possible.
11. Process according to claim 1 or 2, in which gas streams which keep the ceramic pre-shaped items floating during the sintering and are inert at the sinter temperature are used as contact-free supporting materials.
12. Process according to claim 1 or 2, in which a magnetic field which keeps the ceramic pre-shaped items floating during the sintering because of incorporated or attached magnetic constituents is used as contact-free supporting material.

[illegible]

BIRCH, STEWART, KOLASCH & BIRCH, LLP

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**PLEASE NOTE:  
YOU MUST  
COMPLETE THE  
FOLLOWING**

# COMBINED DECLARATION AND POWER OF ATTORNEY FOR PATENT AND DESIGN APPLICATIONS

As a below named inventor, I hereby declare that: my residence, post office address and citizenship are as stated next to my name; that I verily believe that I am the original, first and sole inventor (if only one inventor is named below) or an original, first and joint inventor (if plural inventors are named below) of the subject matter which is claimed and for which a patent is sought on the invention entitled:

**Insert Title:**

## METHOD OF DIMENSIONALLY SINTERING CERAMICS

**Fill in Appropriate  
Information -  
For Use Without  
Specification  
Attached:**

the specification of which is attached hereto. If not attached hereto,  
the specification was filed on August 3, 2001 \_\_\_\_\_ as  
United States Application Number \_\_\_\_\_;  
and amended on August 3, 2001 \_\_\_\_\_ (if applicable) and/or  
the specification was filed on February 4, 2000 \_\_\_\_\_ as PCT  
International Application Number PCT/EP00/00909 \_\_\_\_\_; and was  
amended under PCT Article 19 on January 9, 2001 \_\_\_\_\_ (if applicable).

I hereby state that I have reviewed and understand the contents of the above-identified specification, including the claims, as amended by any amendment referred to above.

I do not know and do not believe the same was ever known or used in the United States of America before my or our invention thereof, or patented or described in any printed publication in any country before my or our invention thereof or more than one year prior to this application, that the same was not in public use or on sale in the United States of America more than one year prior to this application, that the invention has not been patented or made the subject of an inventor's certificate issued before the date of this application in any country foreign to the United States of America on an application filed by me or my legal representative or assigns more than twelve months (six months for designs) prior to this application, and that no application for patent or inventor's certificate on this invention has been filed in any country foreign to the United States of America prior to this application by me or my legal representatives or assigns, except as follows.

I hereby claim foreign priority benefits under Title 35, United States Code, §119(a)-(d) of any foreign application(s) for patent or inventor's certificate listed below and have also identified below any foreign application for patent or inventor's certificate having a filing date before that of the application on which priority is claimed:

Prior Foreign Application(s)

Priority Claimed

<u>199 04 523.2</u>	<u>Germany</u>
(Number)	(Country)

February 4, 1999  
(Month/Day/Year Filed)

☒ Yes      ☐ No

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(Month/Day/Year Filed)

☐ Yes      ☐ No

I hereby claim the benefit under Title 35, United States Code, §119(e) of any United States provisional applications(s) listed below.

(Application Number) \_\_\_\_\_

(Filing Date) \_\_\_\_\_

(Application Number) \_\_\_\_\_

(Filing Date)

All Foreign Applications, if any, for any Patent or Inventor's Certificate Filed More than 12 Months (6 Months for Designs) Prior to the Filing Date of This Application:

Country	Application Number	Date of Filing (Month/Day/Year)
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I hereby claim the benefit under Title 35, United States Code, §120 of any United States and/or PCT application(s) listed below and, insofar as the subject matter of each of the claims of this application is not disclosed in the prior United States and/or PCT application in the manner provided by the first paragraph of Title 35, United States Code, §112, I acknowledge the duty to disclose information which is material to the patentability as defined in Title 37, Code of Federal Regulations, §1.56 which became available between the filing date of the prior application and the national or PCT international filing date of this application.

(Application Number)	(Filing Date)	(Status - patented, pending, abandoned)
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(Application Number)	(Filing Date)	(Status - patented, pending, abandoned)
01/000,000	01/01/2000	patented
02/000,000	02/01/2000	pending
03/000,000	03/01/2000	abandoned

Insert Requested  
Information:  
(if appropriate)

Insert Prior U.S.  
Application(s):  
(if any)

I hereby appoint the following attorneys to prosecute this application and/or an international application based on this application and to transact all business in the Patent and Trademark Office connected therewith and in connection with the resulting patent based on instructions received from the entity who first sent the application papers to the attorneys identified below, unless the inventor(s) or assignee provides said attorneys with a written notice to the contrary:

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I hereby declare that all statements made herein of my own knowledge are true and that all statements made on information and belief are believed to be true; and further that these statements were made with the knowledge that willful false statements and the like so made are punishable by fine or imprisonment, or both, under Section 1001 of Title 18 of the United States Code and that such willful false statements may jeopardize the validity of the application or any patent issued thereon.

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